

times or more than a thousand times as great as that generated by a single-indicator reporter molecule. By increasing signal-to-noise ratios relative to those available from prior reporter compounds, the invention of these reporter compounds

can be expected to facilitate the detection of very small amounts of target compounds—for example, particular genes in blood samples.

This work was done by Wenhong Fan, Jun Li, and Jie Han of Ames Research

Center. Further information is contained in a TSP (see page 1).

Inquiries concerning rights for the commercial use of this invention should be addressed to the Patent Counsel, Ames Research Center, (650) 604-5104. Refer to ARC-14908-1.

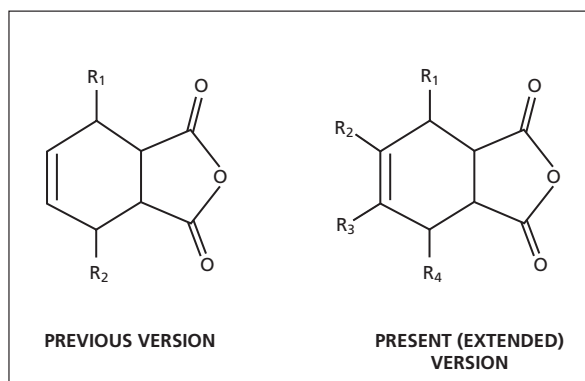
Better End-Cap Processing for Oxidation-Resistant Polyimides

Cross-linking in an inert atmosphere (as opposed to air) yields better results.

John H. Glenn Research Center, Cleveland, Ohio

A class of end-cap compounds that increase the thermo-oxidative stability of polyimides of the polymerization of monomeric reactants (PMR) type has been extended. In addition, an improved processing protocol for this class of end-cap compounds has been invented.

The class of end-cap compounds was described in “End Caps for More Thermo-Oxidative Stability in Polyimides” (LEW-17012), *NASA Tech Briefs*, Vol. 25, No. 10 (October 2001), page 32. To recapitulate: PMR polyimides are often used as matrix resins of high-temperature-resistant composite materials. These end-cap compounds are intended to supplant the norbornene end cap (NE) compound that, heretofore, has served to limit molecular weights during oligomerization and, at high temperatures, to form cross-links that become parts of stable network molecular structures. NE has been important to processability of high-temperature resins because (1) in limiting molecular weights, it enables resins to flow more readily for processing and (2) it does not give off volatile byproducts during final cure and, therefore, enables the production of void-free composite parts. However, with respect to ability of addition polymers to resist oxidation at high temperature, NE has been a “weak link.” Consequently, for example, in order to enable



End Caps of These Molecular Structures are alternatives to previously reported end caps for increasing the thermo-oxidative stability of polyimides. R1 through R4 can be any of a variety of substituents (for example, alkyl, alkoxy, aryl, halogen, or nitro), the inclusion of which reduces the cross-linking temperature.

norbornene-end-capped polyimide matrices to last for lifetimes up to 1,000 hours, it is necessary to limit their use temperatures to $\leq 315^{\circ}\text{C}$.

Like NE, these end caps are also subject to oxidation at high temperatures, but they oxidize in a different manner, such that the long-term stability of a polymer made with one of these end caps exceeds the long-term stability of the corresponding polymer made with NE. Hence, use temperatures and/or lifetimes can be increased. The approach taken in formulating these end caps is to seek derivatives that preserve the desirable processing properties of NE while exploiting one of the modes of the thermo-oxidative degradation of the nadic end cap in such a way as to retard

the overall thermo-oxidative degradation of the affected polymers.

The figure depicts the generic molecular structures of the prior version and the present extended version of this class of end caps. Each end cap is a 1,2,3,6-tetrahydrophthalic anhydride, substituted in such a way as to lower the cross-linking temperature. The end cap maintains its stability during imidization (at 200°C) and cross-linking.

If the imidization is carried out in air, then the end cap subsequently aromatizes in competition with cross-linking. This aromatization is undesirable. Therefore, the improved processing protocol specifies that the process be carried out in an inert atmosphere, wherein cross-linking is the predominant, if not the exclusive, reaction path. Following cross-linking, the end cap is spontaneously converted, upon aging in air, to a thermally stable capping group.

This work was done by Mary Ann B. Meador of Glenn Research Center and Aryeh A. Frimer of Bar Ilan University, Israel. Further information is contained in a TSP (see page 1).

Inquiries concerning rights for the commercial use of this invention should be addressed to NASA Glenn Research Center, Commercial Technology Office, Attn: Steve Fedor, Mail Stop 4-8, 21000 Brookpark Road, Cleveland, Ohio 44135. Refer to LEW-17429.